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Influence of different treatments on the decomposition of Ammonium Heptamolybdate

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The constitution of precursors and their treatment during activation or decomposition can influence the catalytic activity of the products. Important properties which may be affected are composition, crystallinity, particle size and the structure. In this study, the decomposition of ammonium heptamolybdate tetrahydrate (AHM) is studied. The decomposition products are molybdenum oxides with different stoichiometrie and with different constitution. Utilized techniques were in situ X-ray absorption spectroscopy (XAS) and X-ray diffraction (XRD). Additionally, TG/DTA studies were carried out. In all three methods, a simultaneously monitoring of the decomposition products in the gasphase is carried out with a masspectrometer. The influence of different flow rates, atmospheres, mechanical treatment and heating rates were elucidated.

The influence of the different atmospheres were studied with all three techniques, and the experimental conditions were hold as consistent as possible for comparability. The analysis of the data, by comparison of temperatures, formed intermediates and products, show that this purpose is fulfilled. Thus, for the different conditions, extensive information of the decomposition mechanism is obtained. The analysis of the in situ XRD data gave the evolution of phases on the long range order scale and their crystallinity. This information is used as a starting point for the analysis of the EXAFS data, obtained by the XAS measurements. The analysis provide changes on the short range order scale, which can not be detected by XRD. Accordingly, from this complementary methods, the structural evolution can be described completely. The analysis of the NEXAFS data, obtained by the XAS measurements, by means of principle component analysis and examination of the edgeshift, gave additionally information on composition and oxidation state.

Detailed analysis of the influence of heating and gasflow rates with TG/DTA measurements enables to study the kinetics of the decomposition.

In Figure 1a) and b), as an example, the evolution of the XRD pattern and the evolution of the radial distribution (RDF) function with temperature, of the decomposition in helium is shown. There are two features which are directly noticeable by comparing the two evolutions. First is in the temperature range between 370 K and 520 K. Whereas from the XRD pattern it is seen that the intermediate in this temperature range is almost x-ray amorphous and so no structural information can be obtained, in the (RDF) there is still information about the short range order available. Second is in the temperature range between 620 K and 770 K. Here no change upon further heating in the RDF is visible. Whereas on the opposite in the XRD an increase in crystallinity is observable.

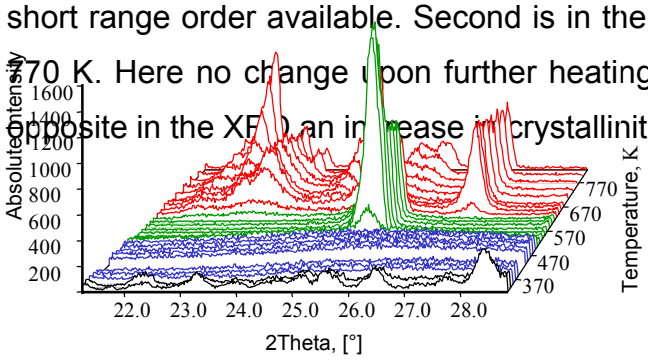

Figure 1a Evolution of XRD pattern with temperature of the AHM decomposition in helium

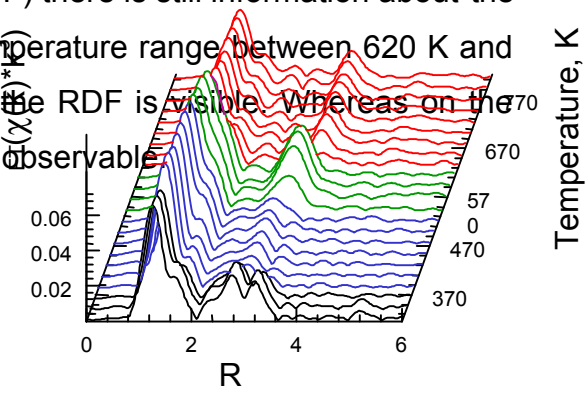

Figure 1b Evolution of the RDF with temperature of the AHM decomposition in

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